

ATTACHMENT NO? CO

4655 BIDDLE AVENUE, WYANDOTTE, MICHIGAN 48192 . (313) 285.9200

rebruary 18, 1981

Mr. Robert J. Courchaine Chief, Water Division Department of Natural Resources Stevens T. Mason Building Box 30028 Lansing, MI 48909



Dear Mr. Courchaine:

As part of the requirements of Section C, Process Waste Characterization Study, of Pennwalt's Final Order of Abatement, a detailed procedure used for the characterization of Process 45 - Triethylamine oxide/Diethylhydroyxlamine, is attached.

A liquid chromatographic method for the analysis of hexadecyl mercaptan and the corresponding disulfide is nearly complete, with the exception of a few minor details.

Since this product is made very infrequently, we are confident that we will have a fully completed method available by the end of the second quarter for the next projected production run.

Attempts at development of a method for Methane Sulfonyl Chloride and Methane Sulfonic Acid have not been nearly as successful. To date, we have been unable to obtain consistent results using the same technology that has been so successful for amines and their derivatives.

These two compounds are so highly polar and acidic that the gas chromatography-purge and trap system utilized for much of the work during the study has so far been unsuccessful.

Liquid chromatography is also complicated by the fact that neither the Methane Sulfonic Acid or the Methane Sulfonyl Chloride is ultra violet active; the use of refractive index detection is both insensitive at the desired levels and unreliable.

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Mr. Robert J. Courcaine .
Chief, Water Division
Department of Natural Resources

ATTACHMENT NO?

-2-

We are currently experimenting with the liquid chromatography of aromatic amine derivatives of Methane Sulfonic Acid and Methane Sulfonyl Chloride, using ion exchange separation techniques, combined with an ultraviolet detector. The results, so far, have been encouraging. We will keep you advised of our progress.

Sincerely,

PENNWALT CORPORATION

♂. E. Rhodes

Manager, Technical Department

cc: Paul Zugger

David Batchelor Roy Schrameck

GC .OCEDURE FOR DIETHYLHYDROXYLAM... IN WATER

SCOPE:

To analyze waste water for DEHA and/or its decomposition products to the lppm level.

APPARATUS:

A CDS (Chemical Data Systems) model 310 trapping concentrator (fitted with their desorber and standard traps) with necessary hardware to mate to the GC used.

GC

Perkin Elmer Sigma I system fitted for on column injection using a 1/4" glass column with split disector flow to FID and NPD. Carrier gas used - Helium at 75 psig.

GC COLUMN

Glass 6 feet x 2mm ID Chromosorb 102 with 7% Triton x 305 and 0.5% KOH (80-100 Mesh)

Syringe: Hamilton CR 700-200

PROCEDURE:

The CDS 310 is mated to the Sigma I by a 2" x 1/8" to 1/16" ss connector. It replaces the GC septum retaining nut, and is connected to the CDS 310 valve assembly discharge with a 1/8" Swagelok tube fitting. Follow the CDS manual for set up of necessary piping of carrier gas and air supply. The CDS system will control the carrier gas.

Set up the GC with the 6 ft. glass column specified above so the column will extend all the way through the GC injection port and seat against a septum inside the CDS connecting adaptor. The CDS parameters are as follows:

Carrier gas 30ml/min. at 75 psig

Desorber flow 40ml/min.

Desorber Temperature - 200°C - Heat 5 minutes - Cool 8 minutes

*Valve Temperature 200°C (approximately)

*CAUTION (refer to the manual on valve operating procedures)

Trap temperature - 200°C - 8 minutes

PROCEDURE (continued)

The Sigma I system procedure is Method #2 (see Attachment #1) and is used with a dual detection arrangement using a detector splitter 50/50 to the FID and NPD.

The column and trap system must be conditioned with repeated injections of the cleanest water obtainable. Use 2ul of water direct through the CDS "column injection port" until a reproducible scan is obtained. (See Attachment #2).

To condition the traps and desorber chamber, inject 10ul of water directly into the desorber chamber and heat for 5 minutes onto trap and cool 8 minutes. (The more water injected the longer the heat and cool cycle will have to be). The trap is then heated for about 6 to 8 minutes at 200°C backflushing onto the column.

Repeat runs until a consistent scan similar to Attachment #3 is obtained. A new column may take two or three days to condition.

Once a good blank run has been obtained, a sample run is first made using 2ul of sample injected directly to the column. Attachment #4 shows a typical scan of a test solution of 52ppm of a fresh DEHA mix through the CDS trap system. As the sample ages it will change to a combination of the peaks at 6.48, 7.90 and 8.37. If the DEHA is about 20-25ppm or less, it will decompose almost completely with the peak at 6.48 being the only one of measurable amounts. If nothing is detected, or very low response using 2ul, then inject up to 10 to 20ul into the desorber and trap system to concentrate and backflush to column.

The method must be calibrated with fresh standards.

FO THE ! PPM LEVEL IN ' "STE WATER.

ATTAChment #1

COL-- GLASS 6FT 2MM ID CHROMOSORB 102 (80-100 mesh)
7% TRITON 305 + 0.5%kOH

L2 LST2 ATTACHMENT NO!

METHOD 2

ANALYZER CONTROL

INJ TEMP 200
DET ZONE 1,2 250 25
BUX TEMP 25
FLOW A.B 30 5
INIT OVEN TEMP, TIME 75 0

TEMP RATE TIME 225 12.0 8

DATA PROC

STD WT.SMP WT 1.0000 1.0000 1
FACTOR,SCALE 1 0
TIMES 20.40 0.00 11.10 14.50 327.67 327.67
SENS-DET RANGE 200 20 0.00 2 0 0
UNK,AIR 1.000 0.00
TOL 0.0000 0.050 1.0
REF PK 0.000 0.00 0.00
STD NAME

EVENT CONTROL

ATTH-CHART-DELAY 3 10 0.01

(QF, 15 2MM ID GLASS 6FT C102-242-7-0,5KOH PUN 28 Red Blank - Direct injection

NPD - Beach 410 Range / ATEA

Supering a paig

FID - Air 30 paig

Nydrogen 26 paig

Nydrogen 26 paig

This is associated with water SENSITIUITIES 200 20 BGN -4.30 6.40 7,42 9.70 12.40 16.35

FILE

47

HHAL

CET

1

MET

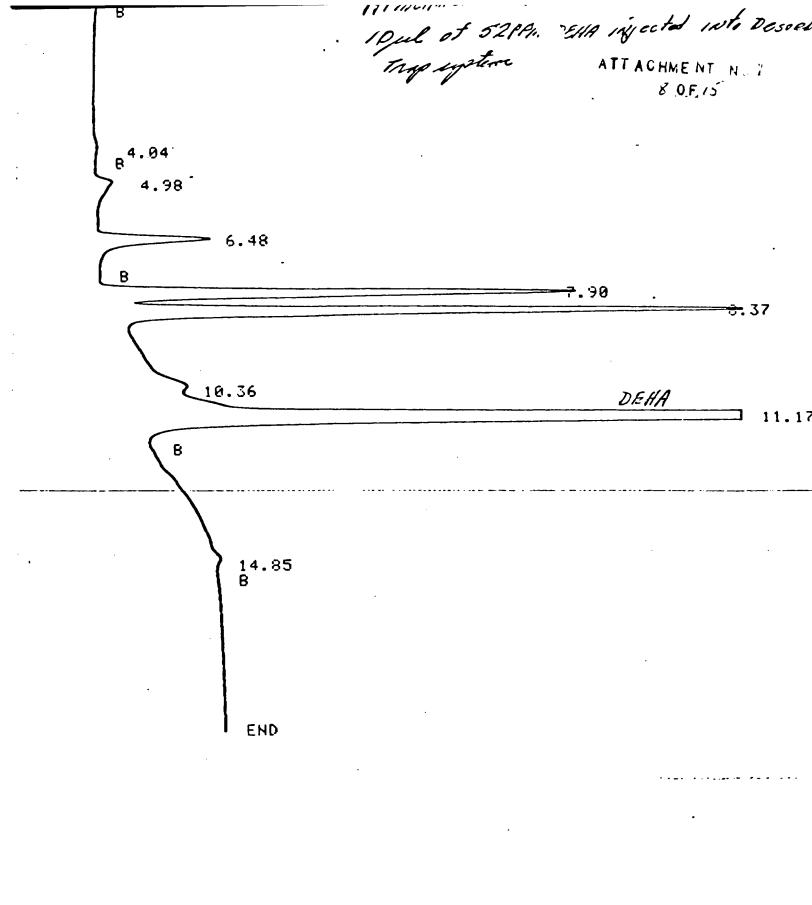
ATTACHMENT : N7 .

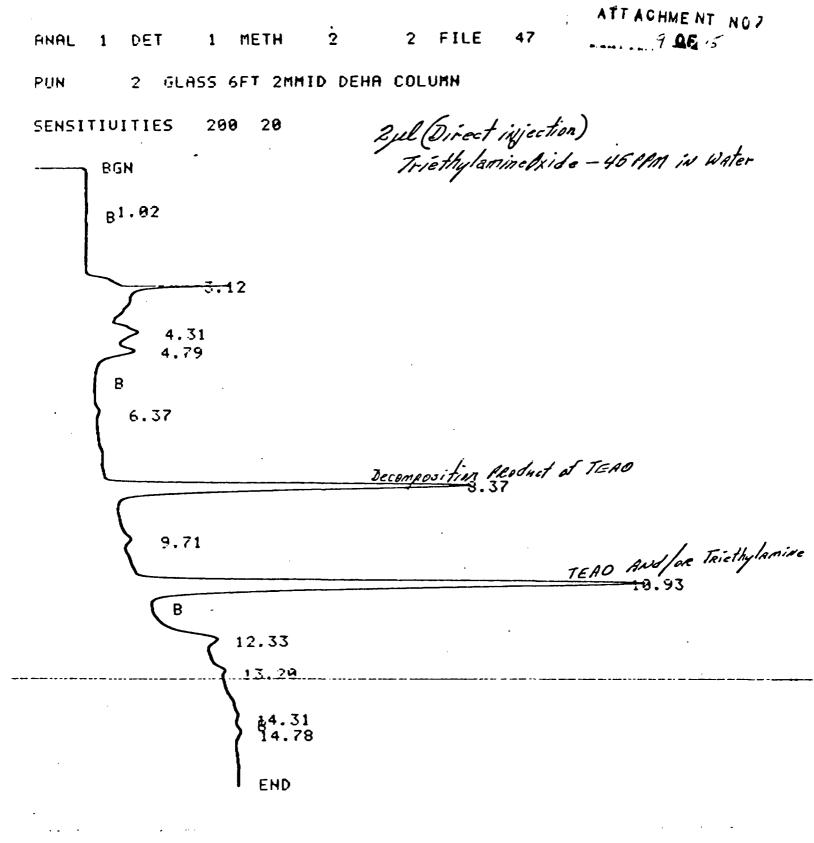
ATTACHMENT NO 7

ATTACHMENT NO7

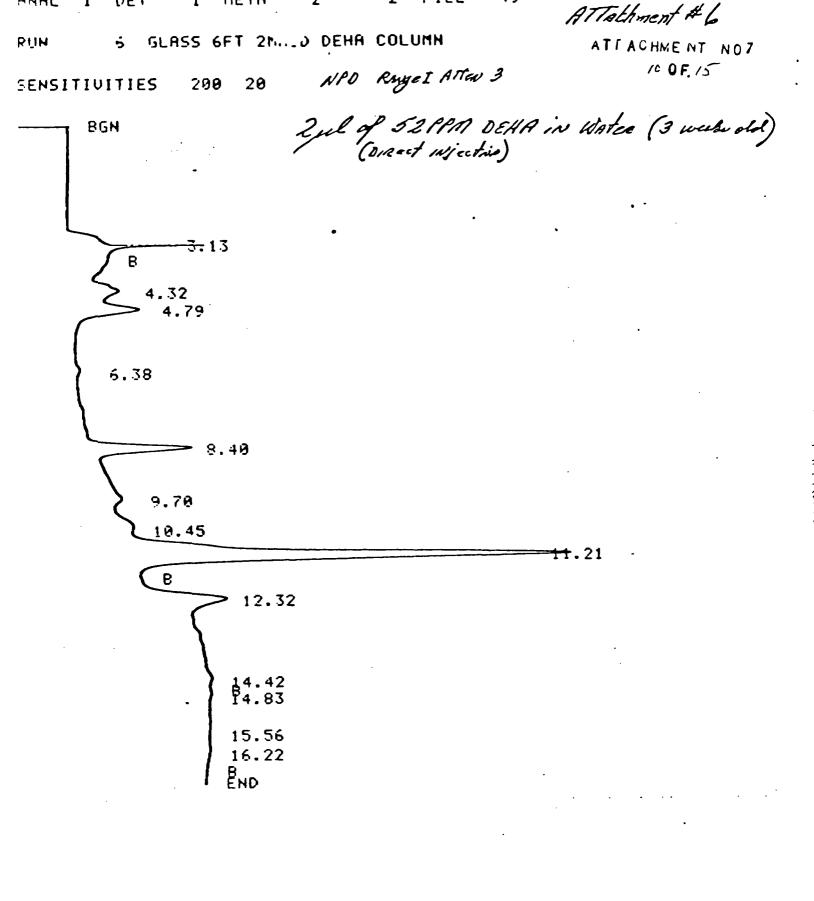
6.36

JEND





HITACHMENT " 2



1 DET

HHAL

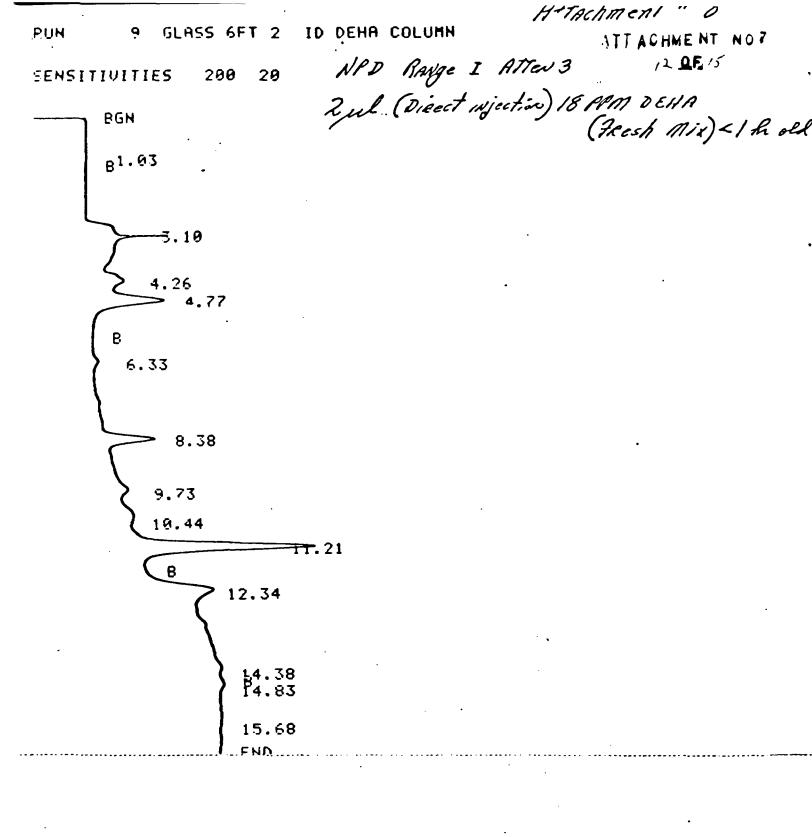
1 MEIH

2

FILE

A+tachment #7 ID DEHA COLUMN PUN GLASS 6FT 1 7 ATTACHMENT NOT NPD Range I ATTEN 3 "I DE 15

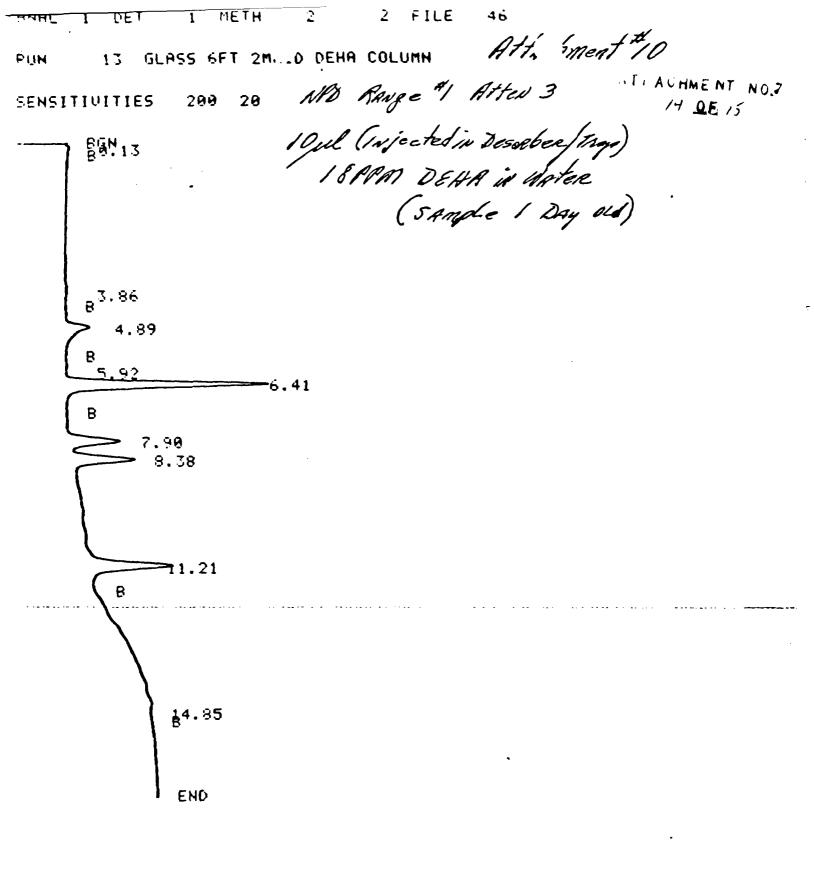
2 Lal (Direct inj) of 52 1 pm DENA (I Day old) 200 20 SENSITIVITIES BGN g1.20 3.09 4.26 6.34 8.36 9.68 H.17 14.85 END



A. Lachment #9 TO DEHA COLUMN PUN GLASS 6FT 2 NPD RANGE Z ATTEN 3 13 NE 15

2 ul (Direct njection) 18 APM DENA

(18 Days old) 298 20 SENSITIVITIES BGN 6.35 8.35 9.70 10.37 12.31 END



SAN	PLING POIN	T:	sot	JTH IN	TAKE	D	ATE	ANA	LYZED				D	ATE R	EPOR	TED	·:		
rin	E OF SAMPL	ING:			_	CO	MPOS	ITE	DATE				GI	RAB D	ATE:				
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2.	mg/l chl			=						4.	C.	0.D	<u> </u>						
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	Results mg/1 Cl	=	ml A	gNO ₃	х	N AgN	03	x	me	W	X	10)6	\dot{z}	m]	Ls	amp 1 e	:	
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	N FeSOz	· (NH	14)2SO	<u> </u>	m]	L K ₂ Cr ₂ (2,	Х	N K ₂	Cr ₂ 0	,	<u>:</u>	m1	FeSO	. (NF	14)	2SO4		and the second
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	ъ. с.о.	D	mg/l																
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<u>3.</u>	mg/l chlori	de			=					6.	mg	3/1	C.O.D	•	_=			
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10⁶

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50

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x 0.008

Page 2 - 001 MONITORING

GRAB	DATE:
ANALYSIS	DATE:

6.	C.O.D Tues. CHEMICAL OXYGE	n demani) - (GRAB - mg/		METHOD :		0340				
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	b. C.O.D mg/ Results mg/1 C.O.D.		ank-	Sample)	1	FeSO4.	x	me	ew X	106	÷	ml sample

X

X

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14	- STANDAR th EDITION ORET - EPA			S OF	WATE	ER A	AND W	ASTE V	VA TEF	A	BOVE	1	. 2.	3	· ·	4.	5.	6.
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$\frac{2.}{3.}$	mg/1 chl			10111	=	_			<u>+</u> +				C.O.D.		=	 		0.52-5
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				=	<u> </u>		х		100	- / -	<u> </u>	÷	100					
3.	Cl - Mon CilORIDE				- m	g/1	1	METHO	D SM	1 - 40	8A							
	Results mg/l Cl			AgNO	T		N Ag		х	me		х	10 ⁶	÷	ml	samp:	le	
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4.	$MH_3 - N - 1$ APPIONIA					POS	SITE -	mg/1		METH	OD ST	ORE	r 00610					
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∤	Spec Resu		noton	<u>etri</u>	c ab	sor	bance	read	ing	=		10	e) or 8 n	1 20	mg	N mplo		
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	b. Titra		n Met	hod											-			
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6.	C - O - D -	See	Page	2												=		

COMPOSITE	DATE:	
ANALYSIS	DATE:	

6.	C.O.D Tue CHEMICAL OXY				OMPOSIT	'E -	mg/l	MET	HOD	STORET 0	340				
	a. Standard	izat	ion:	Nor	mality	of F	eSO4•(NH4)2S	04						
	N FeSO4.	(NH4) ₂ SO ₄	=	ml K ₂	Cr ₂ 0	, X	N K ₂	Cr ₂ 0	, <u>÷</u>	m1	FeSO4.	(NH ₄),	SO ₄	
					25		X			+					
	b. C.O.D mg/1														
	Results mg/l C.O.D.	=	(Blan	nk-Sa	ample)	X	N Fe	50 ₄ .) ₂ 50 ₄	х	mew	Х	10 ⁶	÷	ml sample	
		11	(_)	х			х	0.008	х	10 ⁶	÷	50	
		=				Х			Х	0.008	Х	10 ⁶	÷	50	

S۸	MPLING	POI	IT:	003	WAY.	NE O	<u>co</u> .	J	DATE	ANALY	ZED:_			DA :	re R	EPOF	≀TED:_			
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5.	рН - рН -			'F _	3.00	т и^	<i>D</i> .	TODE	m 00/	.00	_ ,,,	_								
=	- DIL -	<u> </u>	0011		1:5	<u> </u>	2.3	71/7/7	T_004	<u>+</u> <u> </u>	HC	↓_= -↓								

SAN	PLING POINT	LING POINT: 005 POND DATE ANA											DA T	E REP	ORTE	ED:_		
TIN	Œ OF SAMPLI	ING:_				COM	POSIT	E DA'	TE:_				GRA	B DAT	E:			·
TEN	PERATURE:	c	°c	o _F					r	TEST	ED B	Y:	 -	APP	ROVI	ED B	Y:	
14 t	- STANDARD h EDITION			WAT	ER AI	ND WAS	STE W	ATER.	,			1.	2.	3.		4.	5.	6.
	RET - EPA N									ABOV.								
NOT	E: 10 ⁶ = PARAMETER		000,000)		ANSWE		LIM	דידי	· · · · · · · · · · · · · · · · · · ·						ΔΝ	SWER	LIMIT
1.			ed soli	ds =		MOWEI	`	35		4.	mg.	/1 a	mmoni	a as	N =		D N LIK	1.0
$\frac{2}{2}$.	mg/l resi					~			0	5.	pН				=			6.5-
$\frac{2.}{3.}$	mg/l chlo				=					6.	mg,		.O.D.		=			
					71-	77												
1.	S.S Mo SUSPENDED							מטניים	см	_ 20:	g n							
	SUSPENDED	301	TDS -	GRAD	1	3/ I	ME	INOD	514		<u> </u>						···	
İ	Gross wt.	=			1			ľ										
	(-)	$\neg \uparrow$			 				T			T		1		Re	sults	
•	Tare wt.	=			1			,	c l	10 ⁶	-	n	ıl sam	ple	=		/1 s.s	
									$\neg \Gamma$			1		`			· · · · · · · · ·	
	Ppt. wt.	=			<u> </u>				<u> </u>	10 ⁶	<u> </u>		100		=			
2.	Res. Cl ₂	- Da	ilv -	7 day	rs/wl	· · ·					·							
	RESIDUAL						M	ЕТНОІ	o si	4 - 40	09 E							
	a. 1 ml	of	FeSO.	(NH ₄)	2SO	= 10	0 mi	crogi	cams	of	resid	lual	chlo	rine.			~	····
	Resu			<u> </u>	<u></u>	1					1	1						
ı	b. mg/1	Res	. Cl ₂	=	Tit	er	X 1	Micro	ogra	ms/m	1 .	÷l	mls	ample				
																		
				=			X	10	00		:	:	1	00				
3.	Cl - Mon.	.Wed	.Fri.			<u>_</u>						<u>-</u> -						
۱ .	CHLORIDES				mg/1	L	METHO	OD SI	1 -	408A								
	Results							T	Ī			T		l I	1			
1	mg/1 Cl	=	ml Ag	NO ₃	х	N Ag	NO ₃	Х	1	mew	İ	x	10 ⁶	÷	m1	sar	nple	
											[
		=_			Х	0.08	5528	Х	0.	03545	53	Х	106	<u>÷</u>	 	5	·	
4.	NH ₃ -N - M	on.	Wed. F	ri.										<u> </u>	•			
	AMMONIA A				MPOS	SITE -	mg/	1	ME	THOD	STOR	RET	00610					
			e Meth															
	Spect	roph	otomet	ric a	bsor	bance	reac	ling	=	:			=			g N		
	Resul					1				1	-	10	or	8 ml 1	net	samp	le	
	mg/1	NH ₃ -	N =	mg	N	X	1,00	00	<u>÷</u>	400) x 5	00						
	•					_		,	•	1 .								
{			=			X	1,00	10	<u>÷</u>	8	3							
	b. Titra	tion	Metho	d:												,		
	Resul			Ï			T		\neg						1	_		480
1	mg/1 1		N =	ml	H ₂ SO	4 X	N	H ₂ SO	ایا	x	me	W	х	10 ⁶	-	_	400 x	
									\top				-					
]			=			X	0.0	2		x	0.01	401	X	10 ⁶	-		384	
5.	pH _ Daily	,	7 David					-		T -	T							
-								- 1			}							
	pH - COMPO			ETHO!	STO	ORET C	0400		pН	<u> </u>	<u> </u>							
6.	C.O.D. Se	e Pa	age 2		-				-									

COMPOSITE	DATE:
ANALYSIS	DATE:

6.	C.O.D Tue				OMPOSITE	- m	g/1	METH	od s	TORET 0	340				
	a. mg/l Cl	fro	m ste	p 3,	page l =	·		mg/l							
	b. Calcula	tion	of h	ow mu	ch HgSO4	to :	add to	sampl	e:	·····	····				
	1. A	50 m	ıl sam	ple i	s used fo	or C	.O.D.						·		
	2. mg	/1 C	1 📥	20 =	=		÷ 2	20 =	·		mg (Cl in	. 50 π	nl s	ample.
	3. mg	<u>C1</u>	in 50	ml 🛶	- 1000	=		g	rams	Cl in	50 m	l sam	ple		
	4. g	Cl i	n 50 1	m1 x 1	10 =			g HgS	υ, t	o add t	o C.	D.D.	samp]	le	
	En	ough	HgSO,	isa	added to	tak	e care	of 10	x C	l prese	nt.		·	·	
	c. Calcula	tion	of h	ow muc	ch NaCl	to a	dd to	Salt Co	orre	ction S	amp 1	≥:	·		
	1. g	Cl i	n 50 r	nl x 1	1.6485	=		g	NaC	l to ad	l to	Salt	Corr	ect	ion Sample
	2. 1.	6485	=	NaCl	/Cl mol.	wt.	rati	.0							
	d. Standar	diża	tion:	Nor	mality o	of Fe	eSO4.(NH4),SO),						
	N FeSO4.	(NH4) ₂ SO ₄	=	ml K ₂ Cı	207	Х	N K ₂ Cı	207	-	ml l	eSO,	• (NH ₄	sر(04
				=	2 5		Х		-	+					
	c. C.O.D.	on s	ample	befor	e salt o	corre	ection	l :			1				
	mg/1 C.O.D.	=	(Bla	ank 🕶	Sample)	x	NK	2Cr ₂ O ₇	x	me	w)	1	06	<u>_</u>	ml samp
		=	(-)_	х			Х	0.00	3 ,	(1	06	÷	50
		=		•		X			X	0.00	3 >	7	06	-	50
	f. C.O.D.	on sa	alt co	orrect	ion samp	le:					•				
	mg/1 C.O.D.				ample)	x	N Ka	Cr ₂ O ₇	х	mew	х	10 ⁶] ÷	! m	l sample
			(<u></u>	.)	Х		<u> </u>	Х	0.008		10 ⁶	<u>.</u>	* · ·	50
		_				х			X	0.008		10 ⁶	<u>:</u>	;· —	50
	g. Net mg/	L	. n .	n 005	. <u> </u>				^				<u></u>	· –	<u> </u>
	Results mg/1 C.		=	mg/l	C.O.D.		ıncorr	ected	-	mg/1				t co	orrected
			=						-						

LABORATORY REPORT - OUTFALL 006 NPDES MONITORING

Date Sampled: Comp	P:		An	alyzed a	ind Repor	ted:		
" " Grat	o:		Si	gned:	·			
					OUTFALL	006	SOUTH INTAKE	NON- CONTA
PARAMETER	UNITS	METHOD	SAMPLE	AVE.	MAX.	RESULT		
Temp.	°F		-					
Res. Cl ₂	mg/l	SM-409É	Grab		0.5	<u></u>		
Chlorides	mg/1	SM-408A	Comp		147*			
NH ₃ -N	mg/l	EPA-00610	Grab	1.5	3.0			
Sus. Solid	mg/l	SM-208D	Comp.	6.3	9.5*			
COD	mg/l	SM-508	Comp.					
BOD ₅	mg/l	SM-507	Comp.	6.3	9.5	·		
BOD ₅ DATE SAMPLED		-	-					
Pheno1	ug/l	SM-510C	Comp.		200			
Sulfides	mg/1	SM ₁ -228A	Comp.					
			рн снес	CK_				
		LAB _		····				
		RECORDER _		····				
		METER						
		GATEHOUSE _						
	SM = S	Standard Meth	ods, Water	and Wa	ste Wate	r 14th Edit	ion	

SM₁ = Standard Methods, Water and Waste Water 13th Edition

EPA = Environmental Protection Agency Manual 1974 Edition

*NPDES limits are expressed in Net #/Day. Limits shown are based on an average flow of 7.2 MGD. Reported values are on a gross basis.

DISTRIBUTION: B100, E40, G96, G100, K4, L5, M41, M47, P28, R8, S72, Shift Supt. W